

ANNEX 4

183G02 - file 2

Shuttle Report # 183G02
SU010398 capsule

ACCO. USA
WHEELING, ILLINOIS 60090

15044



50505 15044 1
OPEN/VERT. FEUILLE/VERDE SUAVE

Analyses Report

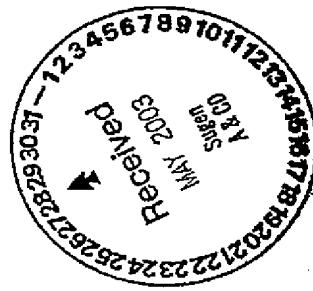
Global Pharmaceutical Sciences - Italy

Submission Id: 117733
Sample ID (Request No.): 20023347
User sample ID (lot): B3G02
Sample Type: SUC10398 (PN1200940A) CAPSULE
MG

**Request Type: STABILITY
Stability Study: IBSG02
Pack Type: 30 ml PLAS
Storage Condition: 25°C/60% RH
Time Label: 18 MONTH
Preparation purpose: STABILITY**

Condition: APPROVED
Status: COMPLETE

Samples Notes:		Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
Method	Controls											
083GCG02#00	CAPSULE APPEARANCE	0=UNCHANGED	1	bd							CAPPUCIO	MACCARI
	CAPSULE COLOR	0=UNCHANGED	1	bd							CAPPUCIO	MACCARI
	CONTENT APPEARANCE	0=UNCHANGED	1	bd							CAPPUCIO	MACCARI
	CONTENT COLOR	0=UNCHANGED	1	bd							CAPPUCIO	MACCARI
	SMELL	0=UNCHANGED	1	bd							CAPPUCIO	MACCARI
083GTD1#01_S	(HPLC TITER) SAMPLE#1	100.0	1	%				90.000	- 110.000	15545/86-JAN 2003	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#2	101.0	1	%				90.000	- 110.000	15545/86-JAN 2003	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#3	100.6	1	%				90.000	- 110.000	15545/86-JAN 2003	CAPPUCIO	MACCARI
	AVERAGE TITER (HPLC)	100.53	1	%				90.000	- 110.000	15545/86-JAN 2003	CAPPUCIO	MACCARI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	0.77	1	%						15545/86-JAN 2003	CAPPUCIO	MACCARI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	1	%						15545/86-JAN 2003	CAPPUCIO	MACCARI
083GKF01#00	(HUMIDITY) SAMPLE #1	1.9	1	%						15545/86-JAN 2003	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #2	1.8	1	%						15545/86-JAN 2003	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #3	1.8	1	%						15545/86-JAN 2003	CAPPUCIO	MACCARI
	AVERAGE HUMIDITY	1.83	1	%						15545/86-JAN 2003	CAPPUCIO	MACCARI



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Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 117733

Sample ID (Request No.): 20023347

User Sample ID (lot): 183G02

Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG

Request Type: STABILITY
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 18 MONTHS
Preparation purpose: STABILITY

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE#1	109.0	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#2	102.2	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#3	106.8	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#4	104.3	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#5	106.5	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#6	105.0	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	DISSOLUTION	105.63	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	2.342	1	std-1				15545/86- JAN 2003	CAPPUCCIO	MACCARI
	C.V.	2.22	1	%				15545/86- JAN 2003	CAPPUCCIO	MACCARI
	MIN. DISSOLUTION VALUE	102.2	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	MAX. DISSOLUTION VALUE	109.0	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI

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Analyses Report

Global Pharmaceutical Sciences - Italy

Submission Id: 117733
Sample ID (Request No.): 20025347
User Sample ID (lot): IR3GC02
Sample Type: SUB10388 (PNU290940AD) CAPSULE 50 MG

Samples Notes:

Observations on the Method

083GC02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method

083GC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.77

RRT

Area %

0.65	0.08
0.89	0.06
0.91	0.10
0.94	0.05
1.06	0.11
1.10	0.06
1.28	0.09
1.29	0.11
1.70	0.11

Request Type: STABILITY
Stability Study: IR3G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 18 MONTHS
Preparation purpose: STABILITY

Condition:
APPROVED
status:
COMPLETE

INTERNAL USE ONLY

SAMPLE APPROVED BY: GIUSEPPE MACCARI
APPROVE DATE: 26-may-2003

Time Points

PHARMACIA

Pharmaceutical Development

Submission Id: 116558

Sample ID (Request No.): 20021516

User Sample ID (lot): 183G02

Sample Date: 31/05/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 50

MG

Stability Study Start Date: 31/05/2001

► S.P.N. 2001005 - Manufacturer PDI/PHARMACIA Nerviano - API Batch
(A)5975-MTM-0002

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#0	SAMPLE#1	92.80	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
1	SAMPLE#2	95.91	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	SAMPLE#3	96.76	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	SAMPLE#4	100.16	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	SAMPLE#5	99.02	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	SAMPLE#6	98.74	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	DISSOLUTION	97.23	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	STANDARD DEVIATION	2.67	std-1				15545/27- JUN 2002	CAPPUCIO	MACCARI
	C.V.	2.75	%				15545/27- JUN 2002	CAPPUCIO	MACCARI
	MIN. DISSOLUTION VALUE	92.80	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
	MAX. DISSOLUTION VALUE	100.16	%		80.000		15545/27- JUN 2002	CAPPUCIO	MACCARI
083GMC01#0	TOTAL AEROBIC BACTERIA	IN LIMITS	bt				15545/27- JUN 2002	CAPPUCIO	MACCARI
1	ESCHERICHIA COLI	ABSENT	bt				15545/27- JUN 2002	CAPPUCIO	MACCARI
	SALMONELLA	ABSENT	bt				15545/27- JUN 2002	CAPPUCIO	MACCARI
	FUNGI	IN LIMITS	bt				15545/27- JUN 2002	CAPPUCIO	MACCARI
	STAPHYLOCOCCUS AUREUS	ABSENT	bt				15545/27- JUN 2002	CAPPUCIO	MACCARI
	ENTEROBACTERIA	IN LIMITS	bt				15545/27- JUN 2002	CAPPUCIO	MACCARI

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Pharmaceutical Development

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Integrated Analytical Bulletin

Submission Id: 116558
Sample ID (Request No.): 20021516

User sample ID (lot): 183G02

Sample Date: 31/05/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 50

MG

Stability Study Start Date: 31/05/2001

S.P.N. 2001005 - Manufacturer PDPHARMACIA Nerviano - AP|Batch
(A)5975-MTM-0002

Samples Notes:

Batch Manufacture Date: 17/05/2001

Stability Study: 183G02

Pack type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: STABILITY

Condition:

APPROVED

Status:

COMPLETE

Observations on the Method

083GC01#00, component: CAPSULE APPEARANCE
Opaque brick red colored shaped hard gelatin capsule containing yellow-orange colored powder

Observations on the Method

083GDS01#01, component: DISSOLUTION
Average dissolution at 15 minutes: 96.05% (interval: 90.25%-101.6%)
Average dissolution at 45 minutes: 98.18% (interval: 91.95%-101.57%)

Observations on the Method

083GMC01#01, component: TOTAL AEROBIC BACTERIA

Total bacteria count < 10 Ufc/g (Report No. 02.9470)

Observations on the Method

083GMC01#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g (Report No. 02.9470)

Observations on the Method

083GMC01#01, component: ESCHERICHIA COLI

Report No. 02.9470

Observations on the Method

083GMC01#01, component: FUNGI

Fungi (Yeast + Molds) < 10 Ufc/g (Report No. 02.9470)

Observations on the Method

083GMC01#01, component: SALMONELLA

Report No. 02.9470

Observations on the Method

083GMC01#01, component: STAPHYLOCOCCUS

AUREUS

Report No. 02.9470

Observations on the Method

083GC01#00, component: IMPURITIES NOT LESS

THAN 0.05%

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MACCARI

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PHARMACIA

Pharmaceutical Development

Laboratory
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Integrated Analytical Bulletin

Submission Id: 116558
Sample ID (Request No.): 20021516

User sample ID (lot): B33G02

Sample Date : 31/05/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer PD/PHARMACIA Nerviano - API Batch
(A)5975-MTM-0002

Samples Notes:

Batch Manufacture Date : 17/05/2001
Stability Study: B33G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Observations on the Method
08:GC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Total correlated substances: 0.80%

RT	Area (%)
0.65	0.10
0.90	0.05
0.91	0.09
0.94	0.05
1.00	99.20 (SU 11248)
1.06	0.14
1.09	0.06
1.28	0.07
1.29	0.11
1.70	0.12

Approved by: MACCARI
Approval date: 21-jun-2002

Pharmacia & Upjohn

Pharmaceutical Development



Analyses Report

Laboratory
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Submission ID: 113751
Sample ID (Request No.): 20011710

User sample ID (lot): **103602**
Sample Type: Solid (PRUZERAMAD) CAPSULE 50
MG.

Samples Notes:

Stability Study: 103602
Pack Type: BULK
Storage Condition: TIME ZERO
Time Label: TIME ZERO
Preparation purpose: STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
036020100	APPEARANCE	COMPLIANT	NG				7535 - JUN 2001	PASTO	MARIANI
036020101	HPLC IDENTIFICATION	POSITIVE	NG				7535 - JUN 2001	PASTO	MARIANI
036020101	(HPLC TITER) SAMPLE#1	91.3	%		96.000	- 105.000	7535 - JUN 2001	PASTO	MARIANI
036020101	(HPLC TITER) SAMPLE#2	97.7	%		95.000	- 105.000	7535 - JUN 2001	PASTO	MARIANI
036020101	(HPLC TITER) SAMPLE#3	94.1	%		95.000	- 105.000	7535 - JUN 2001	PASTO	MARIANI
036020101	AVERAGE TITER (HPLC)	98.03	%		95.000	- 105.000	7535 - JUN 2001	PASTO	MARIANI
036020100	TOTAL CORRELATED SUBSTANCES	1.3	%				7535 - JUN 2001	PASTO	MARIANI
036020100	IMPERITIES NOT LESS THAN 0.05% SEE TEXT		%				7535 - JUN 2001	PASTO	MARIANI
036020100	(HUMIDITY) SAMPLE #1	1.45	%				7535 - JUN 2001	PASTO	MARIANI
036020100	(HUMIDITY) SAMPLE #2	1.65	%				7535 - JUN 2001	PASTO	MARIANI
036020100	(HUMIDITY) SAMPLE #3	1.57	%				7535 - JUN 2001	PASTO	MARIANI
036020100	AVERAGE HUMIDITY	1.59	%				7535 - JUN 2001	PASTO	MARIANI

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Pharmaceutical Development



Analyses Report

Laboratory
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Sample Number: 113751
Sample ID (Request No.): 20011710

User sample ID (lot): 103002

Sample Type: SOLUT/50 (PNU25084040) CAPSULE 50
MG

Stability Study: 103002

Peak Type: BULK

Storage Condition: TIMEZERO

Time Label: TIMEZERO

Preparation purpose: STABILITY

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
1	SAMPLE #1	97.4	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #2	101.2	%		95.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #3	101.3	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #4	100.3	%		95.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #5	102.2	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #6	96.2	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #7	96.4	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #8	97.2	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #9	95.5	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #10	105.8	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #11	97.3	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #12	97.3	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #13	96.2	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #14	103.7	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #15	99.2	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #16	95.7	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #17	95.8	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #18	99.8	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #19	96.3	%		85.000	- 115.000	75355 - JUN 2001	PASTO	MARIANI
	SAMPLE #20	97.5	%					PASTO	

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Pharmaceutical Development

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Analyses Report

Submission Id: 133751

Sample ID (Request No.): 20011710

User sample ID (lot): 080302

Sample Type: SUSTAINED (PULSATILE) CAPSULE 50 MG

Samples Notes:

Stability Study: 180302
Pack Type: BULK
Storage Condition: TIME ZERO
Time Label: TIME ZERO
Preparation purpose: STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
080302010	SAMPLE #20	97.5	%	%	85.000	- 115.000	75355 - JUN 2001		
	AVERAGE WEIGHT	80.61	%	%	86.000	- 115.000	75355 - JUN 2001	PASTO	
	STANDARD DEVIATION	2.922		std-1			75355 - JUN 2001	PASTO	
	C.V.	2.95	%	%			75355 - JUN 2001	PASTO	
	MINIMUM VALUE	85.5	%	%	85.000	- 115.000	75355 - JUN 2001	PASTO	
	MAXIMUM VALUE	105.6	%	%	85.000	- 115.000	75355 - JUN 2001	PASTO	
<hr/>									
0803020110	1.12								
	DESCRIPTION	TOTAL AEROBIC BACTERIA	IN LIMITS	test					
1		ESCHERICHIA COLI	ABSENT	test					
		SALMONELLA	ABSENT	test					
		FUNGI	IN LIMITS	test					
		STAPHYLOCOCCUS AUREUS	ABSENT	test					
		ENTEROBACTERIA	ABSENT	test					

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Pharmaceutical Development

Analyses Report

Laboratory
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Submission Id: 103751

Sample ID (Request No.) : 20011710

User sample ID (lot) : 031622

Sample Type: Sustance (PRE20094040) CAPSULE 50
M3

Stability Study: 103622
Pack Type: BULK
Storage Condition: TIME ZERO
Time Label: TIME ZERO
Preparation purpose: STABILITY

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
03622000	SAMPLE #1	97.5	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
1.12	SAMPLE #2	100.5	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #3	98.0	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #4	95.5	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #5	99.9	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #6	97.2	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #7	98.7	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #8	99.3	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #9	97.8	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #10	98.7	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #11	99.0	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	SAMPLE #12	98.0	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	DISSOLUTION	98.51	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	STANDARD DEVIATION	1.325	SD-1					PASTO	MARIANI
	C.V.	1.36	%					PASTO	MARIANI
	MIN. DISSOLUTION VALUE	85.5	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI
	MAX. DISSOLUTION VALUE	100.5	%		80.000	100.000	7535 - JUN 2001	PASTO	MARIANI



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Pharmaceutical Development

Analyses Report

Laboratory
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Information Id: 113751

Sample ID (Request No.): 20011710

User sample ID (lot): 103G02

Sample Type: SUPPOSITES (PVC/PP/PP/40AD) CAPSULE, E 50

MG

Stability Study: 104602

Pack Type: BULK

Storage Condition: TIMEZERO

Time Label: TIMEZERO

Preparation purpose: STABILITY

Condition:

APPROVED

Status:

COMPLETE

Samples Notes:

Observations on the Method 083GCO1#01, component: APPEARANCE

Opaque brick red colored shaped hard gelatin capsule containing yellow-orange colored powder

Observations on the Method 083GDS01#01_12, component: MAXIMUM DISSOLUTION VALUE

no comment

Observations on the Method 083GDS01#01_12, component: DISSOLUTION

Average dissolution at 15 minutes: 97.7% (C.V. 2.6%)

Average dissolution at 30 minutes: 96.5% (C.V. 1.4%)

Average dissolution at 45 minutes: 99.5% (C.V. 1.7%)

Observations on the Method 083GMC01#01, component: TOTAL AEROBIC BACTERIA

Total bacteria count < 10 Ufc/g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: ESCHERICHIA COLI

Test done on 10g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: FUNGI

Fungi (yeasts and molds) < 10 Ufc/g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: SALMONELLA

Test done on 10g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: STAPHYLOCOCCUS

AUREUS

Report No. 01.1231

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

correlated substances
RRT 0.690 (Impurities A): 0.16%

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Analyses Report

Laboratory
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Submission Id: 113751
Sample ID (Request No.): 20011710

User sample ID (lot): 113602

Sample Type: SUPPOS (PRU2000/00) CAPSULE 50 MG

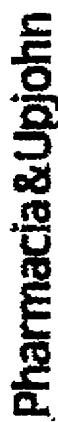
Samples Notes:

Observations on the Method 0836C01#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT 0.913 (IMPURITIES B): 0.00 %
RRT 0.926 (IMPURITIES C): 0.15 %
RRT 1.018 (IMPURITIES D): 0.20 %
RRT 1.039 (IMPURITIES E): 0.11 %
RRT 1.342 (IMPURITIES F): 0.07 %
RRT 1.251 (IMPURITIES G): 0.13 %
RRT 1.272 (IMPURITIES H): 0.15 %
RRT 1.646 (IMPURITIES I): 0.16 %

Observations on the Method 0836C01#01, component: AVERAGE WEIGHT
The average weight is equal to 38.61% which corresponds to 87.86 mg per capsule.

Approved by: MARIAM
Approval date: 13-10-2001



Pharmaceutical Development

Integrated Analytical Bulletin

Laboratory
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Submission Id: 113982

Sample ID (Request No.): 200120014

User sample ID (lot): M34GR2

Sample Date: 30/01/2001

Sample Type: SOLUBLE (PAKUZERBAGAD) CAPSULE 50 MG

Stability Study Start Date: 01/05/2001
S.P.N. 2001005 - Manufacturer: PHARMACHA Mexico - API Batch (ABST3-AU14-0002)

Samples Notes:

Batch Manufacture Date: 17/05/2001
Stability Study: 105302
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/80% U.R.
Time Label: 1 MONTH
Preparation purpose: STABILITYOverall:
APPROVED
Status:
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
0033002100	CAPSULE APPEARANCE	0=UNCHANGED	bd				1544101 - AUG 2001	COLONBOD	MARIAN
0	CAPSULE COLOR	0=UNCHANGED	bd				1544101 - AUG 2001	COLONBOD	MARIAN
	CONTENT APPEARANCE	0=UNCHANGED	bd				1544101 - AUG 2001	COLONBOD	MARIAN
	CONTENT COLOR	0=UNCHANGED	bd				1544101 - AUG 2001	COLONBOD	MARIAN
	SMELL	0=UNCHANGED	bd				1544101 - AUG 2001	COLONBOD	MARIAN
0033001010	(HPLC TITER) SAMPLE#1	94.4	%		80.000	- 110.000	1544102 - AUG 2001	COLONBOD	MARIAN
S	(HPLC TITER) SAMPLE#2	98.1	%		90.000	- 110.000	1544102 - AUG 2001	COLONBOD	MARIAN
	(HPLC TITER) SAMPLE#3	98.0	%		90.000	- 110.000	1544102 - AUG 2001	COLONBOD	MARIAN
	AVERAGE TITER (HPLC)	98.17	%		90.000	- 110.000	1544102 - AUG 2001	COLONBOD	MARIAN
0033001010	TOTAL CORRELATED SUBSTANCES	1.04	%				1544103 - AUG 2001	COLONBOD	MARIAN
0	IMPERITIES NOT LESS THAN 0.05%	SEE TEXT	%				1544103 - AUG 2001	COLONBOD	MARIAN
0033001010	(HUMIDITY) SAMPLE #1	2.0	%				1544103 - AUG 2001	COLONBOD	MARIAN
	(HUMIDITY) SAMPLE #2	2.0	%				1544103 - AUG 2001	COLONBOD	MARIAN
	(HUMIDITY) SAMPLE #3	2.0	%				1544103 - AUG 2001	COLONBOD	MARIAN
	AVERAGE HUMIDITY	2	%				1544103 - AUG 2001	COLONBOD	MARIAN

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Submission ID: 11382
Sample ID (Request No.): 20012049

User sample ID (lot): 083GSC02

Sample Date: 2001/09/01

Sample Type: SURVIVOR (PRINCIPAL) CAPSULE 50
MG

Stability Study Start Date: 2001/09/01
(NBB/75HTM-002)

Samples Notes:

Batch Manufacture Date: 17/05/2001
Stability Study: 083GSC02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% R.H.

Time Label: 1 MONTH

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Observations on the Method 083GSC01#01, component: DISSOLUTION
average dissolution per single capsule after 15 minutes: 96.5% (C.V. 2.0%)
average dissolution per single capsule after 30 minutes: 100.4% (C.V. 1.8%)
average dissolution per single capsule after 45 minutes: 100.7% (C.V. 2.0%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

CORRELATED SUBSTANCES

RRT 0.97%: 0.00%

RRT 0.98%: 0.06%

RRT 1.02%: 0.11%

RRT 1.05%: 0.28%

RRT 1.08%: 0.09%

RRT 1.25%: 0.09%

RRT 1.25%: 0.10%

RRT 1.54%: 0.13%

Approved by: **MARSHAN**
Approval date: 07-Sep-2001

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Submission Id: 113884

Sample ID (Request No.): 20012351

User sample ID (lot): 8335G02

Sample Date: 31/05/2001

Sample Type: 80 IN 1000 (NUC2981000) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001
S.P.N. 201005 - Manufacturer PDP/HARMAFACIA Nucleo - API Batch
(A8975-A114-0002)

Samples Notes:

Method	Control	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
0835G0020	CAPSULE APPEARANCE	0=UNCHANGED	0x				1544102 - AUG 2001	COLOMBOD	MARIANI
	CAPSULE COLOR	0=UNCHANGED	1x				1544102 - AUG 2001	COLOMBOD	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	1x				1544102 - AUG 2001	COLOMBOD	MARIANI
	CONTENT COLOR	0=UNCHANGED	1x				1544102 - AUG 2001	COLOMBOD	MARIANI
	SMELL	0=UNCHANGED	1x				1544102 - AUG 2001	COLOMBOD	MARIANI
0835G00201	(HPLC TITER) SAMPLE#1	97.9	%		90.000	- 110.000	1544102 - AUG 2001	COLOMBOD	MARIANI
S	(HPLC TITER) SAMPLE#2	95.7	%		90.000	- 110.000	1544102 - AUG 2001	COLOMBOD	MARIANI
	(HPLC TITER) SAMPLE#3	98.1	%		90.000	- 110.000	1544102 - AUG 2001	COLOMBOD	MARIANI
	AVERAGE TITER (HPLC)	97.57	%		90.000	- 110.000	1544102 - AUG 2001	COLOMBOD	MARIANI
0835G00200	TOTAL CORRELATED SUBSTANCES	1.21	%				1544103 - AUG 2001	COLOMBOD	MARIANI
	IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%				1544103 - AUG 2001	COLOMBOD	MARIANI
0835G00201	(HUMIDITY) SAMPLE #1	1.9	%				1544103 - AUG 2001	COLOMBOD	MARIANI
	(HUMIDITY) SAMPLE #2	2.1	%				1544103 - AUG 2001	COLOMBOD	MARIANI
	(HUMIDITY) SAMPLE #3	2.0	%				1544103 - AUG 2001	COLOMBOD	MARIANI
	AVERAGE HUMIDITY	2	%				1544103 - AUG 2001	COLOMBOD	MARIANI

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Submission No: 103094

Sample ID (Request No.): 20012051

User sample ID (lot): B32G02

Sample Date: 30/08/2001

Sample Type: 500 (1000) (1042) CAPSULE 50

 H3

Stability Study Start Date: 31/05/2001
S.P.N. 2001005 - Handwritten P/D/PHARMACIA Menlo - API Batch
(ASSET-MTA-002)

Samples Notes:

Batch Manufacture Date: 17/05/2001
Stability Study: 109802
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40°C/75% R.H.
Time Label: 1 MONTH
Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
1	SAMPLE #1	96.9	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	SAMPLE #2	96.7	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	SAMPLE #3	96.0	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	SAMPLE #4	96.9	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	SAMPLE #5	95.2	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	SAMPLE #6	97.5	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	DISOLUTION	97.2	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	STANDARD DEVIATION	1.665	std-1				154414 SEP 2001	PASTO	MARSHAN
	C.V.	1.63	%				154414 SEP 2001	PASTO	MARSHAN
	MIN. DISSOLUTION VALUE	95.2	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN
	MAX. DISSOLUTION VALUE	99.0	%		80.000	90.000	154414 SEP 2001	PASTO	MARSHAN



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Submission Id: 113894

Sample ID (Request No.): 20012051

User sample ID (lot): 1B3G02

Sample Date: 30/05/2001

Sample Type: 500mg (PAN200PHAD) CAPSULE 50
MC

Stability Study Start Date: 31/05/2001
S.P.A. 2001005 - Minidose-Anti PDI/Pharmacia Nephro- - API Batch
(ANP75-MATH-002)

Samples Notes:

Batch Manufacture Date: 27/05/2001

Stability Study: 1B3G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40W/276% U.R.

Time Label: 1 MONTH

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Observations on the Method 083GDS1#01, component: DISSOLUTION
average dissolution per single capsule after 15 minutes: 96.0% (CV 1.5%)
average dissolution per single capsule after 30 minutes: 97.2% (CV 1.6%)
average dissolution per single capsule after 45 minutes: 97.3% (CV 1.6%)

Observations on the Method 083GSC0#00, component: IMPURITIES NOT LESS
THAN 0.05%

CORRELATED SUBSTANCES

RRT 0.891 : 0.05%

RRT 0.916 : 0.05%

RRT 0.928 : 0.13%

RRT 0.936 : 0.05%

RRT 1.004 : 0.37%

RRT 1.008 : 0.10%

RRT 1.260 : 0.03%

RRT 1.270 : 0.15%

RRT 1.655 : 0.44%

Approved by: **MARIANI**
Approval date: 07-sep-2001

PHARMACIA

Pharmaceutical Development

Analyses Report

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Submitted Id: 115782
Sample ID (Request No.) : 20020525

User sample ID (lot) : 183G02

Sample Type: SU01039B (PNU280940AD) CAPSULE 60
MG

Samples Notes:

Request Type: RELEASE
Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

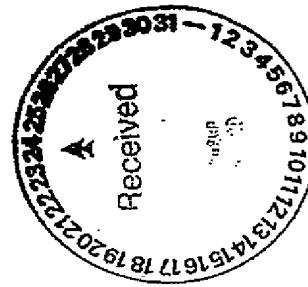
Time Label: 9 MONTHS

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083G002#0	CAPSULE APPEARANCE	CONFORMS	bt				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	CAPSULE COLOR	CONFORMS	bt				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	CONTENT APPEARANCE	CONFORMS	bt				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	CONTENT COLOR	CONFORMS	bt				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	SMELL	CONFORMS	bt				15545/08- MAR 2002	CAPPUCCIO	MARIANI
083GT101#01	(HPLC TITER) SAMPLE#1	100.2	%		90.000	- 110.000	15545/08- MAR 2002	CAPPUCCIO	MARIANI
6	(HPLC TITER) SAMPLE#2	99.5	%		90.000	- 110.000	15545/08- MAR 2002	CAPPUCCIO	MARIANI
	(HPLC TITER) SAMPLE#3	99.4	%		90.000	- 110.000	15545/08- MAR 2002	CAPPUCCIO	MARIANI
	AVERAGE TITER (HPLC)	99.7	%		90.000	- 110.000	15545/08- MAR 2002	CAPPUCCIO	MARIANI
083GSG01#00	TOTAL CORRELATED SUBSTANCES	0.81	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI
083GK#01#00	(HUMIDITY) SAMPLE #1	2.2	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	(HUMIDITY) SAMPLE #2	2.1	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	(HUMIDITY) SAMPLE #3	2.2	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	AVERAGE HUMIDITY	2.17	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI



PHARMACIA

Pharmaceutical Development

Analyses Report

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Submission Id: 115782

Sample ID (Request No.): 20020525

User sample ID (lot): **183G02**

Sample Type: SU010398 (PNU290940AD) CAPSULE 50

MG

Samples Notes:

Request Type: RELEASE
Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE #1	97.15	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	SAMPLE #2	98.05	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	SAMPLE #3	98.61	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	SAMPLE #4	100.49	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	SAMPLE #5	98.57	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	SAMPLE #6	100.12	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	DISSOLUTION	98.17	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	STANDARD DEVIATION	1.291	std-1				15545/08- MAR 2002	CAPPUCIO	MARIANI
	C.V.	1.3	%				15545/08- MAR 2002	CAPPUCIO	MARIANI
	MIN. DISSOLUTION VALUE	97.15	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI
	MAX. DISSOLUTION VALUE	100.49	%		80.000		15545/08- MAR 2002	CAPPUCIO	MARIANI

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PHARMACIA

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Analyses Report

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Submission Id: 115762
Sample ID (Request No.): 20020525
User sample ID (lot): 183G02
Sample Type: SU010398 (PNU1290940AD) CAPSULE 50 MG

Samples Notes:

Observations on the Method 083GCO1#00, component: CAPSULE APPEARANCE
Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS1#01, component: DISSOLUTION
The dissolution values were corrected based on the weight of the respective capsule

Average dissolution at 15 minutes: (interval 96.1% - 100.1%)
Average dissolution at 45 minutes: (interval 98.6% - 100.2%)

Observations on the Method 083GSC1#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.81%

RRT	Area %
0.65	0.08
0.89	0.06
0.91	0.12
0.94	0.05
1.00	98.19 (SU 11248)
1.06	0.20
1.10	0.10
1.26	0.07
1.30	0.13

Approved by: MARIANI
Approval date: 25-mar-2002

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Batch/Job Id: 115149

Sample ID (Request No.): 20013447
User sample ID (lot): NSRG02

Sample Date: 20/11/2001

Sample Type: SOLIDS (PRNUZERTHIAD) CAPSULE 50
MG

Stability Study Start Date: 31/05/2001
SPN: 2001005 - Manufacturer FDY/PHARMACA New Jersey - API Batch
NSRG5/NTA-0002

Batch/Job Id: 115149

Sample ID (Request No.): 20013447
Stability Study: 055302

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/80% U.R.

Time Label: 6 MONTHS

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book/Data Analyses	Analyst	Verifier
0033000100 CAPSULE APPEARANCE	CONFORMS	tot					14327 DEC 2001	CAPPUCIO	MACCARI
CAPSULE COLOR	CONFORMS	tot					14327 DEC 2001	CAPPUCIO	MACCARI
CONTENT APPEARANCE	CONFORMS	tot					14327 DEC 2001	CAPPUCIO	MACCARI
CONTENT COLOR	CONFORMS	tot					14327 DEC 2001	CAPPUCIO	MACCARI
SMELL	CONFORMS	tot					14327 DEC 2001	CAPPUCIO	MACCARI
0033001001 (HPLC TITER) SAMPLE#1	90.57	%			90.000	- 110.000	14327 DEC 2001	CAPPUCIO	MACCARI
(HPLC TITER) SAMPLE#2	98.90	%			90.000	- 110.000	14327 DEC 2001	CAPPUCIO	MACCARI
(HPLC TITER) SAMPLE#3	99.86	%			90.000	- 110.000	14327 DEC 2001	CAPPUCIO	MACCARI
AVERAGE TITER (HPLC)	99.44	%			90.000	- 110.000	14327 DEC 2001	CAPPUCIO	MACCARI
0033001000 TOTAL CORRELATED SUBSTANCES 0.9	%						14327 DEC 2001	CAPPUCIO	MACCARI
IMPURITIES NOT LESS THAN 0.05% SEE TEXT	%						14327 DEC 2001	CAPPUCIO	MACCARI
0033001000 (HUMIDITY) SAMPLE #1	1.7	%					14327 DEC 2001	CAPPUCIO	MACCARI
(HUMIDITY) SAMPLE #2	1.7	%					14327 DEC 2001	CAPPUCIO	MACCARI
(HUMIDITY) SAMPLE #3	1.3	%					14327 DEC 2001	CAPPUCIO	MACCARI
AVERAGE HUMIDITY	1.73	%					14327 DEC 2001	CAPPUCIO	MACCARI



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Substitution Id: 115148

Sample ID (Request No.) 2001347

User sample ID (lot) **MS332**

Sample Date: 30/11/2001

Sample Type: **SPN10006 (PNU20004743) CAPSULE 80**

H8

Stability Study Start Date: 31/05/2001

S.P.N. 2001005 - Manufacturer: POPPHARMACIA Norrland - API Batch:

MS332

Samples Notes:

Batch Manufacture Date : 17/05/2001
Stability Study: 100302
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/90% U.R.
Time Label: 6 MONTHS
Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim.	Upper lim.	Book-Data Analyses	Analyst	Verifier
1	SAMPLE #1	97.9	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #2	100.2	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #3	97.4	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #4	90.8	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #5	102.7	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #6	100.2	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	DISSOLUTION	90.53	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	1.933		std-1			14927 DEC 2001	CAPPUCCIO	MACCARI
	C.V.	1.94	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	MIN. DISSOLUTION VALUE	97.38	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI
	MAX. DISSOLUTION VALUE	102.7	%		80.000	100.000	14927 DEC 2001	CAPPUCCIO	MACCARI

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Sample lot: 116149

Sample ID (Request No.): 20013447

User sample ID (lot): 183G002

Sample Date: 30/11/2001

Sample Type: STUDY (PN122000000/D) CAPSULE 50
MS

Stability Study Start Date: 30/05/2001
S.P.N. 2010/0015 - Manufacturer PDP-FARMACIA Norteno - ACP Brazil
(NORTES-PTM-0012)

Samples Notes:

Observations on the Method 083GC02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Total correlated substances: 0.9%
RRT Area %

0.00	0.005
0.30	0.052
0.31	0.110
1.00	0.096
1.35	0.124
1.27	0.138
1.26	0.110
1.00	0.173
2.17	0.134

Approved by:
MACCABH
11-06-2001

Approval date:

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Laboratory Information Management System

Integrated Analytical Bulletin

Submission Id: 115151
 Sample ID (Request No.): 20013449
 User sample ID (lot): 1801302

Sample Date: 30/11/2001
 Sample Type: SOLID DOSE (PNU/PROD/OD) CAPSULE 50

Sample Study Start Date: 30/11/2001
 S.P.N. 2001/0105 - Manufacturer PNU/PHARMACIA Nürnberg - API Basis
 (A) 0975-0074-0002

Samples Notes:

Batch Manufacture Date: 17/05/2001

Sensory Study: 183602

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% RH.

Time Label: 6 MONTHS

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
0	06360002#0 CAPSULE APPEARANCE	CONFORMS	lit.				14927 DEC 2001	CAPPUCCIO	MACCARI
0	CAPSULE COLOR	CONFORMS	lit.				14927 DEC 2001	CAPPUCCIO	MACCARI
0	CONTENT APPEARANCE	CONFORMS	lit.				14927 DEC 2001	CAPPUCCIO	MACCARI
0	CONTENT COLOR	CONFORMS	lit.				14927 DEC 2001	CAPPUCCIO	MACCARI
0	SMELL	CONFORMS	lit.				14927 DEC 2001	CAPPUCCIO	MACCARI
5	06360001 (HPLC TITER) SAMPLE#1 (HPLC TITER) SAMPLE#2	98.57 98.57	%	%	90.000 - 110.000	110.000	14927 DEC 2001	CAPPUCCIO	MACCARI
5	(HPLC TITER) SAMPLE#3	100.00	%		90.000 - 110.000	110.000	14927 DEC 2001	CAPPUCCIO	MACCARI
5	AVERAGE TITER (HPLC)	99.41	%		90.000 - 110.000	110.000	14927 DEC 2001	CAPPUCCIO	MACCARI
0	06360001#0 TOTAL CORRELATED SUBSTANCES	0.97	%				14927 DEC 2001	CAPPUCCIO	MACCARI
0	IMPURITIES NOT LESS THAN 0.05% SEE TEXT	%	%				14927 DEC 2001	CAPPUCCIO	MACCARI
	06360011#0 (HUMIDITY) SAMPLE #1 (HUMIDITY) SAMPLE #2	2.2 2.2	%	%			14927 DEC 2001	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #3	2.2	%				14927 DEC 2001	CAPPUCCIO	MACCARI
	AVERAGE HUMIDITY	2.2	%				14927 DEC 2001	CAPPUCCIO	MACCARI



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Submission Id: 115151

Sample ID (Request No.): 2001349

User sample ID (lot#): 833G02

Sample Date: 30/12/2001

Sample Type: SUB30826 (PMU200004) CAPSULE 60 MG

Stability Study Start Date: 31/05/2001
SOPN: 2001006 - Mandrelum PDR/HARASAKA Newtendo - API Batch (A8975-NTM-002)

Samples Notes:

Batch Manufacture Date: 17/05/2001

Stability Study: 169002

Test Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/70% RH.

Time Label: 6 MONTHS

Preparation purpose: STABILITY

Condition:

APPROVED

Status :

COMPLETE

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.917%

RT

Area %

0.96	0.08
0.90	0.05
0.91	0.13
1.00	99.03
1.05	0.13
1.08	0.05
1.27	0.14
1.28	0.11
1.49	0.12
2.17	0.12

Approved by: MACCARI
Approval date: 11-dec-2001

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Submission Id: 114395
Sample ID (Request No.) : 20012835
User sample ID (lot) : 163G02

Sample Date : 31/08/2001
Sample Type: SU00388 (PNU280940AD) CAPSULE 50 MG

Stability Study Start Date : 31/08/2001

S.P.N. 200105 - Manufacturer PD/PHARMACIA Nefano - API Batch

(A)S975-NTM-0082

Samples Notes:

083GC00210 CAPSULE APPEARANCE

0=UNCHANGED

1=UNCHANGED

2=UNCHANGED

3=UNCHANGED

4=UNCHANGED

5=UNCHANGED

6=UNCHANGED

7=UNCHANGED

8=UNCHANGED

9=UNCHANGED

10=UNCHANGED

11=UNCHANGED

12=UNCHANGED

13=UNCHANGED

14=UNCHANGED

15=UNCHANGED

16=UNCHANGED

17=UNCHANGED

18=UNCHANGED

19=UNCHANGED

20=UNCHANGED

21=UNCHANGED

22=UNCHANGED

23=UNCHANGED

24=UNCHANGED

25=UNCHANGED

26=UNCHANGED

27=UNCHANGED

28=UNCHANGED

29=UNCHANGED

30=UNCHANGED

31=UNCHANGED

32=UNCHANGED

33=UNCHANGED

34=UNCHANGED

35=UNCHANGED

36=UNCHANGED

37=UNCHANGED

38=UNCHANGED

39=UNCHANGED

40=UNCHANGED

41=UNCHANGED

42=UNCHANGED

43=UNCHANGED

44=UNCHANGED

45=UNCHANGED

46=UNCHANGED

47=UNCHANGED

48=UNCHANGED

49=UNCHANGED

50=UNCHANGED

Condition:

APPROVED

Status:

COMPLETE

Batch Manufacture Date: 17/05/2001

Stability Study: 163G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 3 MONTHS

Preparation purpose: STABILITY

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083G00210	CAPSULE APPEARANCE	0=UNCHANGED	WT		1544127 SEP 2001			PASTO	MARIANI
0	CAPSULE COLOR	0=UNCHANGED	WT		1544127 SEP 2001			PASTO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	WT		1544127 SEP 2001			PASTO	MARIANI
	CONTENT COLOR	0=UNCHANGED	WT		1544127 SEP 2001			PASTO	MARIANI
	SMELL	0=UNCHANGED	WT		1544127 SEP 2001			PASTO	MARIANI
083G01101	(HPLC TITER) SAMPLE#1	99.1	%	90.000	-	110.000	1544127 SEP 2001	PASTO	MARIANI
	(HPLC TITER) SAMPLE#2	100.1	%	90.000	-	110.000	1544127 SEP 2001	PASTO	MARIANI
083G01102	(HPLC TITER) SAMPLE#3	100.6	%	90.000	-	110.000	1544127 SEP 2001	PASTO	MARIANI
	AVERAGE TITER (HPLC)	90.93	%	90.000	-	110.000	1544127 SEP 2001	PASTO	MARIANI
083G001400	TOTAL CORRELATED SUBSTANCES 0.96	%			1544127 SEP 2001			PASTO	MARIANI
	IMPURITIES NOT LESS THAN 0.05% SEE TEXT	%			1544127 SEP 2001			PASTO	MARIANI
083GK01100	(HUMIDITY) SAMPLE #1	1.5	%				1544127 SEP 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #2	1.9	%				1544127 SEP 2001	PASTO	MARIANI
083GK01101	(HUMIDITY) SAMPLE #3	1.4	%				1544127 SEP 2001	PASTO	MARIANI
	AVERAGE HUMIDITY	1.6	%				1544127 SEP 2001	PASTO	MARIANI

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Submission Id: 114395

Sample ID (Request No.): 20012535
User sample ID (lot): 183G02

Sample Date: 31/08/2001

Sample Type: SU010398 (PNU280940AD) CAPSULE 50
MG

Stability Study Start Date: 31/05/2001

Batch Manufacture Date: 17/05/2001
Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Trace Label: 3 MONTHS

Preparation purpose: STABILITY

S.P.N. 2001005 - Manufacturer PDI/FARMACIA NERARIO - API Batch
IA5975-MTM-002

Samples Notes:

Observations on the Method 083GCD02#00, component: CAPSULE APPEARANCE
Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

average dissolution per single capsule after 15 minutes: 96.4% (C.V. 1.5%)

average dissolution per single capsule after 30 minutes: 96.4% (C.V. 1.8%)

average dissolution per single capsule after 45 minutes: 98.6% (C.V. 1.6%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS

THAN 0.05%

Correlated substances

RRT 0.883 : 0.10%

RRT 0.915 : 0.05%

RRT 0.927 : 0.13%

RRT 1.059 : 0.17%

RRT 1.092 : 0.08%

RRT 1.284 : 0.09%

RRT 1.285 : 0.20%

RRT 1.635 : 0.14%

Approved by:
MARIANI

Approval date:
01-oct-2001



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Submission Id: 114397
Sample ID (Request No.) : 20012537

User sample ID (lot) : 183G02

Sample Date : 31/08/2001

Sample Type: SU010398 (PNU280840AD) CAPSULE 50 MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer PDI/PHARMACIA Nerviano - API Batch

(A5975-MTM-0002)

Samples Notes:

Method

Controls

Results

M.U.

Spec?

Lower lim

Upper lim

Book-Data Analyses

Analyst

Verifier

083G0200 CAPSULE APPEARANCE	0=UNCHANGED	txt		15441/27 SEP 2001	PASTO	MARIANI
CAPSULE COLOR	0=UNCHANGED	txt		15441/27 SEP 2001	PASTO	MARIANI
CONTENT APPEARANCE	0=UNCHANGED	txt		15441/27 SEP 2001	PASTO	MARIANI
CONTENT COLOR	0=UNCHANGED	txt		15441/27 SEP 2001	PASTO	MARIANI
SMELL	0=UNCHANGED	txt		15441/27 SEP 2001	PASTO	MARIANI
083G01001 (HPLC TITER) SAMPLE#1		99.6	%	90.000 - 110.000	15441/27 SEP 2001	PASTO
S (HPLC TITER) SAMPLE#2		99.5	%	90.000 - 110.000	15441/27 SEP 2001	PASTO
(HPLC TITER) SAMPLE#3		99.2	%	90.000 - 110.000	15441/27 SEP 2001	PASTO
AVERAGE TITER (HPLC)		99.43	%	90.000 - 110.000	15441/27 SEP 2001	PASTO
083GSC01#00 TOTAL CORRELATED SUBSTANCES 1.07		%		15441/27 SEP 2001	PASTO	MARIANI
IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%		15441/27 SEP 2001	PASTO	MARIANI
083GKF01#00 (HUMIDITY) SAMPLE #1		1.9	%	15441/27 SEP 2001	PASTO	MARIANI
(HUMIDITY) SAMPLE #2		2.1	%	15441/27 SEP 2001	PASTO	MARIANI
(HUMIDITY) SAMPLE #3		2.0	%	15441/27 SEP 2001	PASTO	MARIANI
AVERAGE HUMIDITY		2	%	15441/27 SEP 2001	PASTO	MARIANI

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Submission Id: 114397

Sample ID (Request No.) : 20012537

User sample ID (lot) : 183 G02

Sample Date : 31/08/2001

Sample Type: SU010398 (PNU298840AD) CAPSULE 50 MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer PDI/PHARMACIA Novartis - API Batch (A)5575-ATM-0002

Samples Notes:

Observations on the Method

083GSCO0240, component: CAPSULE APPEARANCE
Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method

083GDS01#01, component: DISSOLUTION

average dissolution single determination after 15 minutes: 95.6% (C.V. 2.7%)

average dissolution single determination after 30 minutes: 96.4% (C.V. 2.7%)

average dissolution single determination after 45 minutes: 96.4% (C.V. 2.7%)

Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Correlated substances

RRT 0.685: 0.11%

RRT 0.915: 0.07%

RRT 0.925: 0.14%

RRT 0.855: 0.07%

RRT 1.055: 0.16%

RRT 1.080: 0.08%

RRT 1.280: 0.08%

RRT 1.292: 0.21%

RRT 1.632: 0.14%

Approved by: MARIANI
Approval date: 01-Oct-2001